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54. Name of Invention:
Fluorine Containing Compounds and Their Manufacturing Methods (Processes)

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Patent Law No. 30, Item 1 applies. In view of announced documents in Showa 60 (1985), November 23 Corporate Juridical Persons' meeting of Chemical Society of Japan, [Showa 60 Chemical Society of Japan, Chugoku, Shikoku Branch Corporate Juridical Persons' Meeting].

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DETAILED DESCRIPTIONS

1. Name of Invention

Fluorine containing compounds and their manufacturing methods (processes).

2. Patent Claims

1. General formula [I]:

$$R'O(CH_2)m\ OR \qquad [I]$$

indicates the fluorine containing compound(s), where R' is a perfluoroalkenyl group, R is a hydrogen atom or a perfluoroalkenyl group, and m is any integer equals or larger than 4.

2. R' is a dimer or a trimer of hexafluoropropane in the fluorine containing compound of Claim 1.

3. R is a hydrogen atom in the fluorine containing compound of Claim 1.

4. R is a dimer or a trimer of hexafluoropropane in the fluorine containing compound of Claim 1.

5. m equals to 4-10 in the fluorine containing compound of Claim 1.

6. General formula [II]:

$$R'F \qquad [II]$$

indicates the perfluoroalkynes, where R' is a perfluoroalkenyl group, and general formula [III]:

$$HO(CH_2)m\ OH \qquad [III]$$

indicates a alkylene alcohol, where m is any integers equal to or larger than 4. The manufacturing process in which compounds [II] and [III] react in a non-aqueous solvent, in the presence of basic catalyst, to give the fluorine containing compound as indicated by general formula [I]:

$$R'O(CH_2)m\ OR$$

where R′ is a perfluoroalkenyl group, R is a hydrogen or a perfluoroalkenyl group, and m is any integer equals to or larger than 4.

7. R′ is a dimer or a trimer of hexafluoropropane in the manufacturing process of Claim 6.

8. R is a hydrogen atom in the manufacturing process of Claim 6.

9. R is a dimer or a trimer of hexafluoropropane in the manufacturing process of Claim 6.

10. m is any integer equal to or larger than 4.

11. Acetonitrile, N,N′dimethylformamide, and THF are the non-aqueous solvent in the manufacturing process of Claim 6.

12. The basic catalyst can be selected from triethylamine, trimethylamine, potassium carbonate, and sodium carbonate in the manufacturing process of Claim 6.

13. The reaction in the manufacturing process of Claim 6 is carried out at 0-25°C.

3. Detailed Explanation of the Invention

Fields of Industrial Applications

This invention concerns new fluorine containing ether compound(s) and its manufacturing process. The fluorine containing compounds can be used as the raw materials for surfactants, water and oil repellents, and fluorine containing oils.

Conventional Technologies

Compounds with perfluorocarbon chain have be actively developed in recent years as the raw materials for surfactants, water and oil repellents, and fluorine containing oils. But the compounds shown in Formula [1] of this invention have not been proposed before; and there has not been any proposal which allows us to obtain such compounds efficiently and with high purity.

The Purpose of This Invention

The objective of this invention is to provide new fluorine containing compounds that can be used as the raw materials for surfactants, water and oil repellents, as well as fluorine containing oils; the objective of this invention is also to provide manufacturing process for such compounds.

The Structure of (the Compounds of) this Invention

The fluorine containing compounds of this invention can be represented by general formula [I]:

$$R'O\ (CH_2)m\ OR \qquad [I]$$

where R′ is a perfluoroalkenyl group, R is hydrogen or perfluoroalkenyl group, and m is any integer equals to or larger than 4. Furthermore, the perfluoroalkyne can be represented by the general formula [II] of this invention:

$$R'F \qquad [II]$$

where R′ is a perfluoroalkenyl group, and alkylene glycol can be represented by the general formula [III]:

$$HO\ (CH_2)m\ OH \qquad [III]$$

where m is any integer equals to or larger than 4. The manufacturing process is as follows: compounds [II] and [III] are used to react in a non-aqueous solvent, in the presence of a basic catalyst, to produce the fluorine containing compound(s) as represented by the general formula [I]:

$$R'O\ (CH_2)m\ OR \qquad [I]$$

where R′ is a perfluoroalkenyl group, R is a hydrogen or a perfluoroalkenyl group, and m is any integer equals to or larger than 4.

The compounds, shown in the general formula [I] of this invention, are such that R′ is a perfluoroalkenyl, tetrafluoroethylene, or hexafluoropropane group or groups derived from their polymers. With tetrafluoroehtylene oligomers (for examples, 2-20 unit polymers), in general, a mixture of several polymers is obtained, and depending on the degree of polymerization, they are difficult to separate, and are used as a mixture. Therefore, it is not always suitable to be used as pure compound (in place where pure compound is required). On the other hand, the oligomers obtained from hexafluoropropane are primarily 2-4 unit polymers, usually dimers and trimers. Thus R′ is a group derived from dimers or trimers of hexafluoropropane.

R is a hydrogen or a perfluoroalkenyl group. In the case where it is a hydrogen, because hydroxyl groups are formed in the compound, they can react with various compounds. For example, it can react with the alkylene oxide group of ethylene oxide, or propylne oxide, etc., to form non-ionic surfactants; it can also react with sulfuric acid and esterizing to form sulfuric acid ester type negative ion surfactants. Isocyanate group can also be introduced into the polymer, for example, as the fluorine containing compounds for surface improvement agent for paints, or for

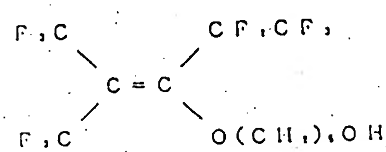
When R is a hydrogen in the compounds represented by the general formula [I], one should use 2 or more equivalents of alkylene glycols for each equivalent of perfluoroalkene (R'F), preferably using 2-3 equivalents, to achieve high yield. Using such equivalent ratio can effectively suppress the formation of R'O (CH_2)m OR', which is very difficult to separate; the excess allylene glycols can be removed from the system by washing with water.

On the other hand, if R is a perfluoroalkenyl group, large excess of perfluoroalkene should be used. In other words, for each equivalent of alkylene glycol, 2 or more, preferably 2-3, equivalents of perfluoroalkene should be used. By doing so, the complete esterification of the end groups of the alkylene glycols can be easily accomplished. If the amount of the alkylene glycols is less than 2 equivalents, a mixture of R'O (CH_2)m OR' and R'O (CH_2)m OH will be formed. They are extremely difficult to separate. Therefore, it is very difficult to obtain the pure compounds.

The following application examples give a more detailed explanation of this invention.

Application Example 1

Synthesis of:



Commercially available Grade 1 reagents, hexamethylene glycol (0.50 mol), triethylamine (0.25 mol), and 100 ml of acetonitrile were placed in a 1000 ml flask equipped with a stirrer, a thermometer, a reflux condenser, and a dropping funnel. The flask was cooled by ice cold water, while perfluoro-2-methyl-2-pentene (one of the dimers of hexafluoropropane) (0.25 mol) was slowly added in drops. After completion of dripping, it was allowed to stir continuously for approximately 8 hours; after the reaction was completed, it was thoroughly washed with a dilute hydrochloric solution, and again repeatedly with water, after which the oily component was dried with anhydrous magnesium sulfate. Area ratio of gas chromatogram confirmed that the yield of the target alcohol was 60 %. By-products, compounds with both end perfluoroalkenyl groups blocked were held to 12 %. The intended product was then easily obtained by fractional distillation. The product was subjected to separation purification by silica-gel chromatography (developing solution: carbon tetrachloride-ethyl acetate 1:1). The isolated fluorine-containing alcohol was analyzed by gas chromatography under the following conditions, a single peak was confirmed.

esterification with acrylic acids or mathacrylic acids. Or, it can be used to partially urethanized polycyanates and used as urethane resin improvement agent. It can also be used to produce fluorine containing oils by esterification of fluorine containing carboxylic acids, or long aliphatic acids that can be used as resin mixing (kneading) improvement agent.

Again, R can be a perfluoroalkenyl group. In general, in this case, R′ can either be the same or a different group. Suitable perfluoroalkenyl groups include, tetrafluoroethylene or its oligomers (for example, 2-20 unit polymers), and hexafluoropropane or its oligomers. Preferably, especially the dimers and trimers of hexafluoropropane. When R is perfluoroalkenyl group, it is especially useful as fluorine containing oils.

m is any integer equals or larger than 4, preferably 4-?0, and most preferably 4-10. If m is smaller than 4, namely, 2 or 3, ring formation reaction will occur preferentially.

The fluorine containing compounds of this invention can be expressed by the following general formula [II]:

$$R'F \qquad [II]$$

where R′ is a perfluoroakenyl group. The perfluoroalkenes can be expressed by the following general formula [III]:

$$HO(CH_2)m\ OH \qquad [III]$$

where m is any integer equals to or larger than 4. Alkylene glycols can be obtained by reacting compounds [II] and [III] in a non-aqueous solvent, in the presence of a basic catalyst.

Examples of R′F include tetrafluoroethylene or its oligomers, and hexafluoropropane or its oligomers. The dimers or trimers of hexafluoropropane are preferred.

Polyalkylene glycols include butylene glycols, pentylene glycols, hexylene glycols, heptyne (heptene?) glycols, octylene glycols, and decene glycols, etc..

The non-aqueous solvents for the reaction of perfluoroalkenes and alkylene glycols include, for example, acetonitrile, N,N′-dimethylformamide, tetrahydrofuran, diglyme, tetrahydropyran, dimethylcarbitol, and dimethyl ether, etc.. Acetonitrile, N,N′-dimethylformamide, and tetrahydrofuran are the preferred solvents.

The basic catalysts used in the reaction promote the removal of hydrogen fluoride, producing and absorbing the water soluble hydrogen fluoride. Examples of the preferred catalysts include triethylamine, trimethylamine, potassium carbonate, and sodium carbonate, etc.. The ratio of the basic catalysts to perfluoroalkene glycols is 2-1 equivalents to 1, usually equal equivalents are preferred.

The reaction is usually carried out under atmospheric pressure and at room temperature, preferably 0-25°C.

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 170°C
Developing temperature	: 120°C
Jet feeding speed	: 20 mm/minute
Retention (holding) time	: 1.50 minutes
Appearance	: colorless transparent liquid (at room temperature)

Infra Red Absorption Spectrum

Absorption based on OH radical (bond)	: 3633, 3350 cm^{-1}
Absorption based on CH radical	: 2933, 2870 cm^{-1}
Absorption based on C=C radical	: 1630 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material HMDS)

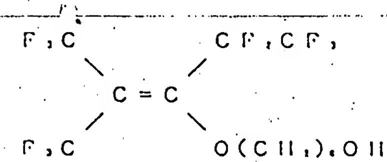
1.49	(multiplet	6 H)
3.50	(multiplet	2 H)
3.96	(singlet	1 H)
4.20	(triplet J = 5.8 Hz	2 H)

^{19}F-NMR Analysis (δ ppm, CFCl was used as internal standard material, high magnetic field side is positive)

56.1	(singlet	3 F)
59.1	(singlet	3 F)
80.5	(singlet	3 F)
113.0	(singlet	2 F)

Application Example 2

Synthesis of:



Commercially available Grade 1 reagents, hexamethylene glycol (0.50 mol), triethylamine (0.25 mol), and 100 ml of acetonitrile were placed in a 1000 ml flask equipped with a stirrer, a thermometer, a f reflux condenser, and a dropping funnel. The flask was cooled with ice cold water, while perfluoro-2-methyl-2-pentene (one of the dimers of hexafluoropropane) (0.25 mol) was slowly added by drops. After com-

pletion of dripping, it was allowed to stir continuously for approximately 8 hours. After completion of the reaction, it was washed thoroughly with a dilute hydrochloric acid solution, and again repeatedly with water. After which the oily component was dried with anhydrous magnesium sulfate. Area ratio of chromatogram confirmed that the yield of the target alcohol was 71 %. By-products, compounds with both end perfluoroalkenyl groups blocked were held to 8 %. The dried oily component was fractional distilled to obtain the target compound with a boiling point of 88.0°C at 2.5 mm Hg. The fraction was analyzed with gas chromatography under the following conditions. A single peak was confirmed.

Gas Chromatography Conditions

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 170°C
Developing temperature	: 120°C
Jet feeding speed	: 20 mm/minute
Retention time	: 2.2 minutes
Appearance	: colorless transparent liquid (at room temperature)
Boiling point	: 88.0°C at 2.5 mm Hg

Infra Red Absorption Spectrum

Absorption based on OH radical	: 3650, 3325 cm^{-1}
Absorption based on CH radical	: 2947, 2865 cm^{-1}
Absorption based on C=C radical	: 1633 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material HMDS)

1.41 (multiplet 8 H)
3.50 (triplet J = 6.0 Hz 2 H)
4.22 (triplet J = 6.2 Hz 2 H)
~~4.46 (singlet 1 H)~~

^{19}F-NMR Analysis (δ ppm, CFCl was used as internal standard material, high magnetic field side is positive)

55.8 (singlet 2 F)
59.0 (singlet 3 F)
80.1 (singlet 3 F)
112.5 (singlet 2 F)

Application Example 3

Synthesis of $C_9F_{17}O(CH_2)_4OH$:

Except pentamethylene glycol was replaced by butylene glycol, and hexafluoropropane dimer was replaced by a trimer, otherwise similar to Application Example 1 was carried out and obtained a colorless transparent liquid (at room temperature) with a boiling point of 69-71°C, at 1 mm Hg. The yield was 55 %. The analytical results are as follows:

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 300°C
Developing temperature	: 70-250°C (temperature increase at a rate of 20°C/minute)
Retention time	: 4.00 minutes

Infra Red Absorption Spectrum

Absorption based on OH radical	: 3600-3050 cm^{-1}
Absorption based on CH radical	: 2956, 2890 cm^{-1}
Absorption based on C=C radical	: 1662 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material TMS)

1.63	(multiplet 4 H)
2.20	(singlet 1 H) *1
3.55	(triplet J = 5.5 Hz 2 H)
3.93	(triplet J = 5.5 Hz 2 H)

Determined in a 10 % by weight CCl_4 solution

*1 disappeared with addition of D_2O

Application Example 4

Synthesis of $C_9F_{17}O(CH_2)_5OH$:

Except the hexafluoropropane dimer was replaced with a trimer, other conditions remained the same as in Application Example 1, and obtained the above compound. The yield was 21 %.

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 300°C
Developing temperature	: 70-250°C (temperature increase at a rate of 20°C/minute)
Retention time	: 3.53 minute
Appearance	: Colorless transparent liquid (at room temperature)
Boiling point	: 108-109°C at 4 mm Hg

Infra Red Absorption Spectrum

Absorption based on OH radical	: 3600, 3000 cm^{-1}
Absorption based on CH radical	: 2950, 2878 cm^{-1}
Absorption based on C=C radical	: 1614 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material TMS)

1.47	(multiplet		6 H)	
2.10	(singlet		1 H) *2	
3.52	(triplet	J = 5.5 Hz	2 H)	
3.87	(triplet	J = 6 Hz	2 H)	

Determined in a 10 % by weight CCl_4 solution
*2 disappeared with addition of D_2O.

Application Example 5

Synthesis of $C_9F_{17}O(CH_2)_6OH$:

Except pentamthylene glycols was replaced by hexylene glycol, and hexafluoropropane dimer was substituted with a trimer, other conditions remained the same as in Application Example 1, and obtained a colorless transparent liquid (at room temperature) with a boiling point of 106-110°C. The yield was 50 %. The purified material has the following analyzed values:

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 300°C
Developing temperature	: 75-250°C
Retention time	: 4.48 minute

Infra Red Absorption Spectrum

Absorption based on OH radical	: 3600-3100 cm^{-1}
Absorption based on CH radical	: 2944, 2872 cm^{-1}
Absorption based on C=C radical	: 1614 cm^{-1}
Absorption based on CF radical	: 1360-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material TMS)

1.38	(multiplet		8 H)
2.05	(singlet		1 H) *3
3.55	(triplet	J = 6 Hz	2 H)
3.86	(triplet	J = 9 Hz	2 H)

Determined in a 10 % by weight CCl_4 solution
*3 disappeared with addition of D_2O

Application Example 6

Synthesis of $C_9F_{17}O(CH_2)_{10}OH$:

Except pentamethylene glycol was replaced by decene glycol, and hexafluoropropane dimer was substituted with a trimer, the other conditions remained the same as in Application Example 1, and obtained a colorless transparent liquid with a boiling point of 114-115°C, at 0.12 mm Hg. The analyzed values are as follows:

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 300°C
Developing temperature	: 70-250°C (temperature increase at a rate of 20°C/minute)
Retention time	: 6.88 minutes

Infra Red Absorption Spectrum

Absorption based on OH radical	:	3600-3050 cm^{-1}
Absorption based on CH radical	:	3938-2866 cm^{-1}
Absorption based on C=C radical	:	1614 cm^{-1}
Absorption based on CF radical	:	1400-1100 cm^{-1}

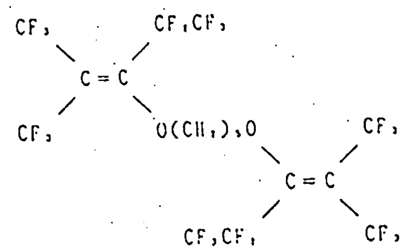
^{1}H-NMR Analysis (δ ppm, internal standard material TMS)

1.27	(multiplet		16 H)
2.00	(singlet		1 H) *4
3.50	(triplet	J = 5.5 Hz	2 H)
3.87	(triplet	J = 6 Hz	2 H)

Determined in a 10 % by weight CCl_4 solution
*4 disappeared with addition of D_2O

Application Example 7

Synthesis of:



Commercially available Grade 1 reagents pentamethylene glycol (0.125 mol), triethylamine (0.25 mol), were dissolved in acetonitrile (100ml) in a 1000 ml flask equipped with a stirrer, a thermometer, a reflux condenser, and a dropping funnel. The flask was cooled with ice cold water. Perfluoror-2-methyl-2-pentene (one of the dimers of hexafluoropropane) was added slowly by drops, and allowed to stir continuously for approximately 8 hours. After the completion of the reaction, it was washed thoroughly with a dilute hydrochloric acid solution; and again repeatedly with water. After which the oily component was dried with anhydrous magnesium sulfate. Gas chromatography area ratio indicated that the yield was 51.3 %. Fluorine containing by-products were held to 1.3 %. The intended product was easily obtained by fractional distillation. The product was then put through separation purification by silica-gel chromatography (developing solution carbon tetrachloride-ethyl acetate 1:1). The isolated fluorine containing compound was subjected to gas chromatography analysis, and a single peak was confirmed.

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier gas	: N_2
Injection temperature	: 170°C
Developing temperature	: 120°C
Jet feeding speed	: 10 mm/minute
Retention time	: 1.87 minutes
Appearance	: Colorless transparent liquid (at room temperature)

Infra Red Absorption Spectrum

Absorption based on CH radical	: 2981, 2925, 2875 cm^{-1}
Absorption based on C=C radical	: 1637 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material HMDS)

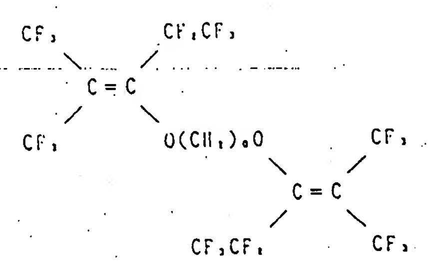
1.60	(multiplet	6 H)
4.17	(multiplet	4 H)

^{19}F-NMR Aanlysis (δ ppm, $CFCl_3$ was used as internal standard material, high magnetic field side is positive)

56.2	(singlet	6 F)
59.2	(singlet	6 F)
80.5	(singlet	6 F)
113.0	(singlet	4 F)

Application Example 8

Synthesis of:



Commercially available Grade 1 reagents hexamethylene glycol (0.125 mol) and potassium carbonate (0.130 mol) and acetonitrile (50 ml) were placed in a 500 ml flask equipped with a stirrer, a thermometer, a reflux condenser, and a dropping funnel. While the flask was cooled with ice cold water, perfluoro-2-methyl-2-pentene (one of the dimers of

hexafluropropane) (0.250 mol) was slowly added by drops. It was allowed to continue to stir for 12 hours. After completion of the reaction, it was washed thoroughly with a dilute hydrochloric acid solution, and again repeatedly with water. After which the oily component was dried with anhydrous magnesium sulfate. Area ratio of gas chromatography confirmed that the yield of the intended fluorine containing ester was 71.2 %. By-product, the fluorine containing alcohol was held to 7.6 %. The intended product was easily obtained by fractional distillation. The product was then isolated by purification through silica-gel chromatography (developing solution, carbon tetrachloride). The Isolated fluorine containing compound was analyzed by gas chromatography under the following conditions, confirming a single peak.

Conditions for Gas Chromatography

Detector	: FID
Column (stainless steel)	: 1 m long, with SE-30 packing
Carrier Gas	: N_2
Injection temperature	: 170°C
Developing temperature	: 120°C
Jet feeding speed	: 20 mm/minute
Retention time	: 3.1 minutes
Appearance	: colorless transparent liquid (at room temperature)

Infra Red Absorption Spectrum

Absorption based on CH radical	: 2950, 2865 cm^{-1}
Absorption based on C=C radical	: 1633 cm^{-1}
Absorption based on CF radical	: 1400-1100 cm^{-1}

^{1}H-NMR Analysis (δ ppm, internal standard material HMDS)

1.50 (multiplet 8 H)
4.20 (triplet 6.2 Hz 4 H)

^{19}F-NMR Analysis (δ ppm, $CFCl_3$ was used as internal standard material, high magnetic field side is positive)

56.0 (singlet 6 F)
59.0 (singlet 6 F)
80.4 (singlet 6 F)
112.7 (singlet 4 F)

Comparison Example 1

Commercially available Grade 1 reagents hexamethylene glycol, $HO(CH_2)_6OH$ (1.00 mol), acetonitrile (200 ml), and

triethylamine (1.00 mol) were placed in a 1500 ml flask, in the same manner as in Application Example 2. While the flask was cooled with ice cold water, perfluoro-2-methyl-2-pentene (one of the dimers of hexafluoropropane) (1.00 mol) was slowly added by dropping. After completion of addition, it was allowed to continue to stir for approximately 8 hours. After completion of the reaction, similar post treatments were carried out as in Application Example 2.

With such equal molar reactants, the yield for fluorine containing alcohol was 45.9 %, and that of the fluorine containing ether was 30.0 %, in other words, there was more of the fluorine containing alcohol by product than the intended fluorine containing ether.

The Effectiveness of this Invention

The compounds of this invention are new compounds that can be used as raw material for fluorine containing surfactants, paints, and modifiers for molding articles, etc.. Furthermore, the manufacturing process of this invention allows one to obtain the compounds of this invention with high yield and high purity.

Patent Applyee: Neos Kabushiki Gaisha (Neos Co. Ltd.)

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